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Synthesis and characterization of a new magnetic composite $MnFe_2O_4$/clay based on a natural clay obtained from Turkestan deposit

The work is devoted to the development of a new method for the synthesis of magnetic composites based on manganese ferrite on a natural clay, coupling with their physico-chemical characterization. In the study, a natural clay of Kazakhstan obtained from the Turkestan deposit was used for the preparation of magnetic composites. The formation of materials with magnetic properties is an urgent task of our time, due to the needs of various applications of magnetically controlled materials for biomedical systems, electronic devices, catalytic and adsorption processes. The advantage of such materials is the ability to control them using a magnetic field for shaking, recovery, induction heating, among others. In this work, samples were prepared by co-precipitation of manganese and iron salts with 5 mol L^{-1} NaOH over the Turkestan clay (TC). Materials were characterized by various analyses, such as Fourier-Transform infrared spectroscopy (FTIR), X-ray diffractometric analysis (XRD), and elemental analysis. According to the results of physical and chemical studies of the XRD and thermal analysis, kaolinite is the main mineral in the composition of TC. Magnetic adsorbents $MnFe_2O_4$/clay with perfect magnetic separation characteristics were successfully obtained by chemical co-precipitation.

Keywords: natural clays, magnetic material, manganese ferrite, adsorbent, modified composite, metal ions, chemical co-deposition, adsorption.

Introduction

Natural clays are inexpensive and readily available materials that work as excellent cation exchangers. The adsorption capacity of clays is due to the relatively high surface area and the net negative charge in their structure, which attracts and holds cations such as heavy metals [1–4]. Natural clays can also be modified to produce materials with enhanced properties, such as metallic pillared clays with improved textural characteristics [5]. Main applications include the action as catalytic materials (taking advantage of the catalytic properties of the incorporated metals) in advanced oxidation processes, such as catalytic wet peroxide oxidation, as well as for removing organic pollutants from contaminated waters [6–8]. The application of metallic particles with magnetic characteristics dispersed on the surface of clays is interesting for the development of magnetic materials that can be magnetically controlled for diverse applications, such as biomedical systems, electronic devices, catalytic and adsorption processes. Magnetic materials can be separated from the medium by a simple magnetic process in catalytic and adsorption processes. Therefore, there is a growing interest in low-cost materials with a high surface area, such as clays, due to their unique applications, including adsorption and catalysis [9].

There are several methods for the preparation such magnetic materials, as magnetite (Fe_3O_4) nanoparticles [10]. These are impregnation, sol-gel, solution combustion synthesis and co-precipitation. Among these methods, chemical co-precipitation is the most promising since it is simple and does not require special chemicals and procedures [11–13].

In this paper, we propose to study a sample of clay from the Turkestan fields. The clays were modified and the physical and chemical characteristics of the natural and modified clay were studied.

Experimental

Materials and reagents

The clays studied were obtained from the Turkestan deposit in Kazakhstan. Sodium hydroxide (NaOH, purity ≥0.97), iron(III) sulfate nonahydrate ($Fe_2(SO_4)_3{\cdot}9H_2O$, purity ≥ 0.98), manganese chloride tetrahydrate ($MnCl_2{\cdot}4H_2O$, purity ≥ 0.99), nickel sulfate heptahydrate ($NiSO_4{\cdot}7H_2O$, purity ≥ 0.99) were used. Initial solutions (50 mg L^{-1}) containing Ni(II) ions were obtained by dissolving pre-determined amounts of nickel sulfate heptahydrate in distilled water. It is worth noting that all the chemicals in this study were used without further purification.

Magnetic materials synthesis

Natural Turkestan clay (TC) was ground into powder in a mill and sorted through a sieve № 0.063, followed by storage in anti-wet bottles at room temperature. To prepare the magnetic composite $MnFe_2O_4$/TC, an aqueous solution containing manganese(II) chloride and iron (III) sulphate in a molar ratio of 1:2 was prepared. Then, 5 g of the sieved TC were added to the Mn(II)-Fe(III) solution, followed by mixing. Afterwards, the co-precipitation of Mn(II)-Fe(III) was conducted by the addition of NaOH solution (5 mol L^{-1}) until pH 10 was obtained. The resulting solution was stirred for 30 min on a magnetic stirrer at room temperature. Then, the suspension was heated to 95-100 °C. After cooling, the prepared magnetic composite was repeatedly washed with distilled water at 50 °C for 2 h. Using a simple magnetic procedure, the resulting materials were separated from the water and dried in an oven at 105 °C for 2 h, to be completely dehydrated and ready for use [14].

Characterization

To characterize the materials, various analyses were conducted. FTIR spectra of the natural and magnetic modified clay were obtained with a FTIR instrument (Infraspec, Model FSM 2202, Russia, St-Petersburg) with a resolution of 1 cm^{-1} and a scan range of 5000 to 500 cm^{-1} using a sample based on 1 % of clay with KBr. X-ray diffractometric analysis was carried out on an automated DRON-3 diffractometer with $Cu_{K\alpha}$-radiation, and a β-filter. Conditions for shooting diffractograms: U = 35 kV; I = 20 mA; shooting θ-2θ; detector 2 deg/min. The results of the elemental composition of natural clays were obtained by using EMP analysis.

Semi-quantitative X-ray diffractomeric analysis was performed applying diffractograms of powder samples by the method of equal attachments and artificial mixtures. The quantitative ratios of the crystal phases have been determined. Interpretation of the diffractograms was carried out using data from the International Centre for Diffraction Data (ICDD) card file; these were the PDF2 Powder diffraction database (Powder Diffraction File) and diffractograms of minerals free of impurities. Shooting conditions: DRON diffractometer — 3.0; accelerating voltage — 35 kV; anode current — 20mA.

Results and Discussion

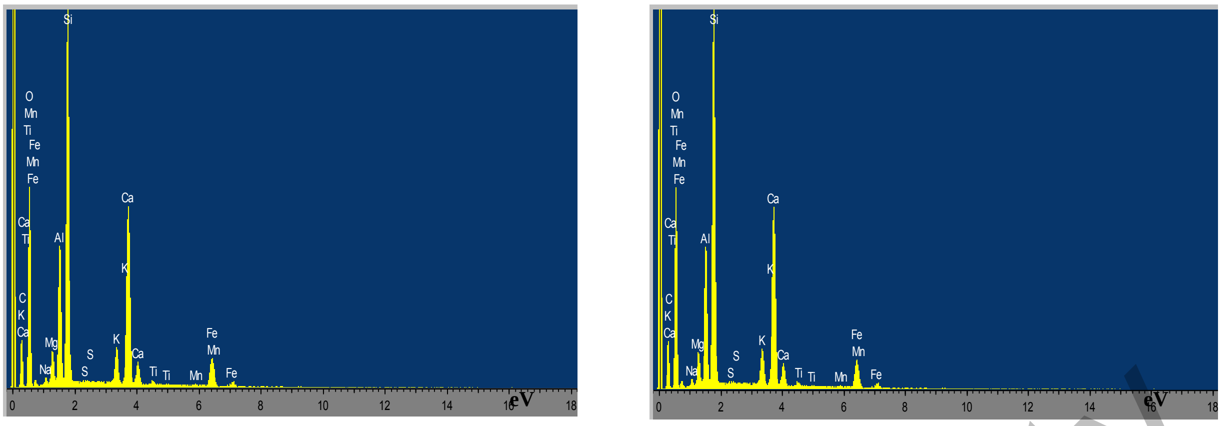
The results of the elemental composition of natural and modified clay were obtained using an electron microprobe (EMP) of the brand Superprobe 733 from JEOL coupled to a spectrometer Inca Energy from Oxford instruments (Figure 1). Table 1 shows the contents of elements in the natural and modified clay. As can be observed, the clay obtained in Turkestan has mainly aluminium (7.12 %), calcium (10.69 %), and silicium (18.97 %) metals (Table 1). Comparing this natural clay with others reported in the literature [15, 16], it is highlighted as positive features the high contents on iron, paramount for catalytic purposes, and on calcium, which allows the preparation of catalytic advanced materials such as pillared clays. The modified clay by co-precipitation also presents high concentration of those metals: aluminium (3.94 %), calcium (7.35 %), and silicium (10.40 %). However, the concentration of all of them decreased after the modification of TC by co-precipitation, whereas the content in iron and manganese strongly increased when compared to the concentration of TC, as expected. Specifically, the composition of the modified clay is rich in iron (22.37 %). In the natural TC, the quantity of Mn is 0.14 %, whereas in $MnFe_2O_4$/TC is 9.23 %. The results also show that the content of Fe increases in $MnFe_2O_4$/TC in comparison with the natural clay.

The magnetic characteristics of the developed material were confirmed using a magnet. Figure 2 is demonstrates an illustrative photo where the magnetic characteristics of $MnFe_2O_4$/TC are placed in evidence upon reaction to a magnetic field.

Table 1

Elemental composition

Materials	Weight of the element (%)										
	O	Na	Mg	Al	Si	S	K	Ca	Ti	Mn	Fe
TC	53.40	0.51	2.20	7.12	18.97	0.06	2.41	10.69	0.31	0.14	4.18
$MnFe_2O_4$/ TC	43.55	0.32	1.24	3.94	10.40	0.17	1.26	7.35	0.19	9.23	22.37



a

b

a — natural TC; *b* — $MnFe_2O_4$/TC

Figure 1. Elemental analysis

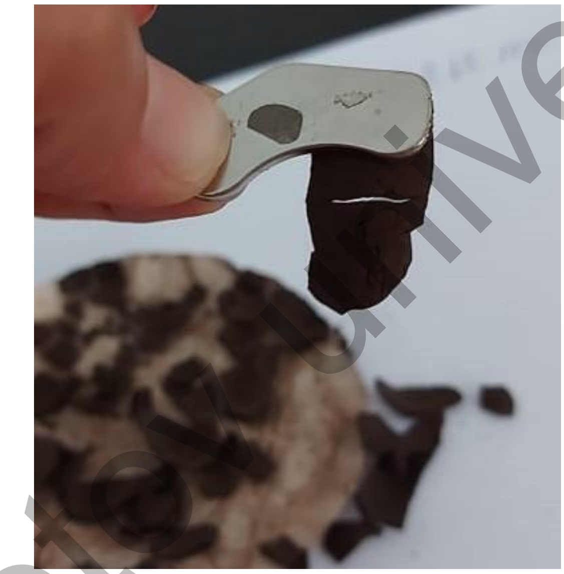


Figure 2. The magnetic characteristics of $MnFe_2O_4$/TC placed in evidence upon reaction to a magnetic field

To determine the quantitative ratio of the crystalline phases of the clay, the samples were submitted to X-ray diffractometric analysis. Possible impurities, the identification of which cannot be unambiguous due to the small contents and the presence of only 1-2 diffraction reflexes, the lack of chemical composition data, or poor crystallization, are indicated in Table 2.

Diffractograms of the sample of natural clay and $MnFe_2O_4$/TC were carried out on an automated diffractometer DRON-3, β-filter. Terms and Conditions caring.com not responsible for diffractograms: U = 35 kV; I = 20 mA; shooting θ-2θ; detector 2 degree/minutes (Fig. 3).

The result of the analysis established that the sample of the studied Turkestan clay belongs to the group of layered silicates-kaolinite $Al_2(Si_2O_5)(OH)_4$, with a low amount of chlorite $(Mg, Fe)_5Al(Si_3Al)O_{10}(OH)_8$, admixtures of muscovite $KAl_2(AlSi_3O_{10})(OH)_2$ and albite $Na(AlSi_3O_8)$**.**

In the modified $MnFe_2O_4$/TC sample, the concentration of minerals increases, but the calcite concentration decreased from 29.9 to 21.4. It is worth noting that the modified vehicle does not contain the mineral kaolinite $Al_2(Si_2O_5)(OH)_4$, however, the analysis showed the presence of the mineral dolomite $CaMg(CO_3)_2$ (Table 2). The characteristic peaks of $MnFe_2O_4$ were also identified, confirming the magnetic characteristics of the developed materials.

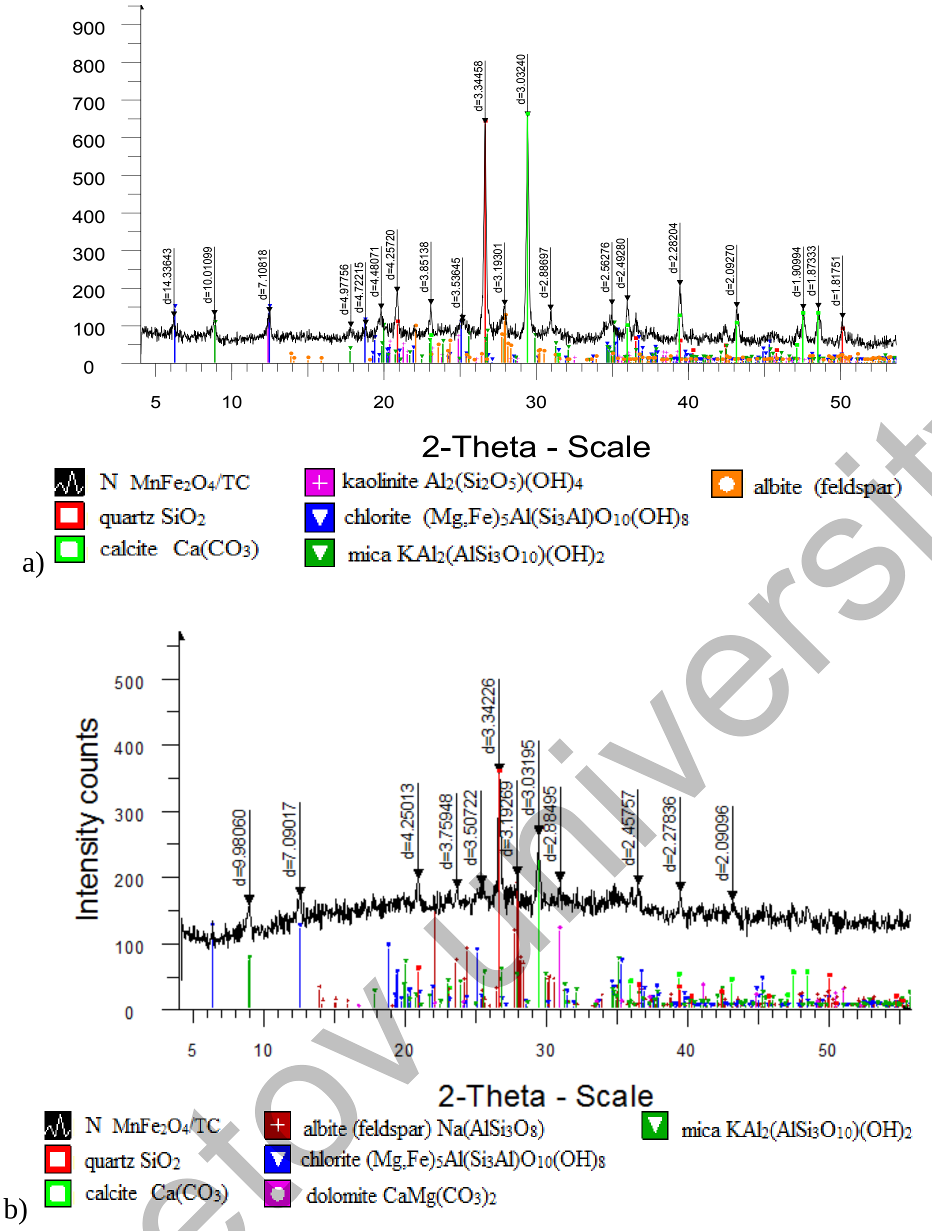


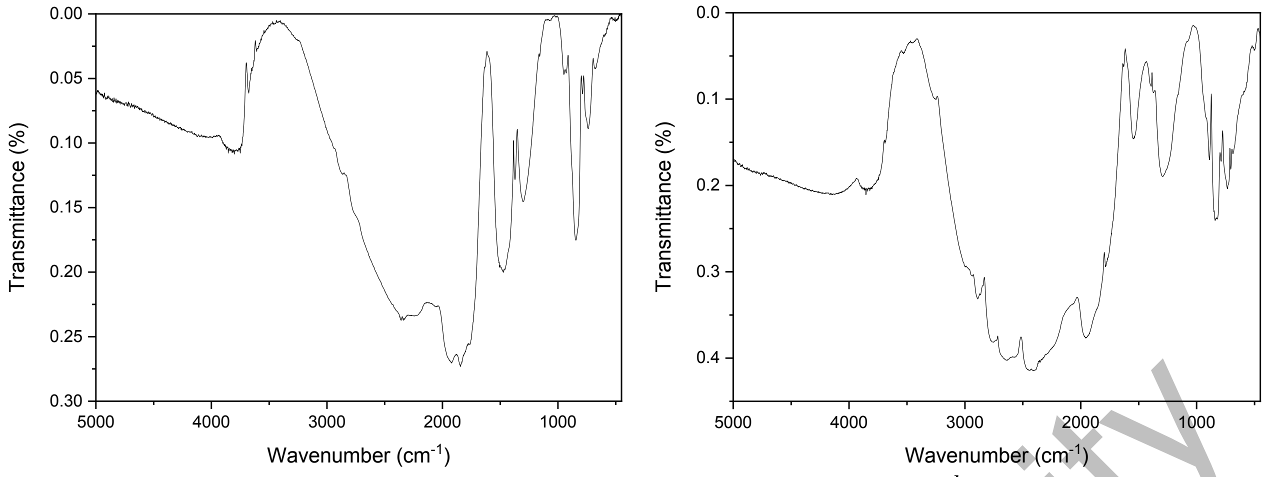
Figure 3. (*a*) Diffractogram of the TC and, (*b*) Diffractogram of $MnFe_2O_4$/TC

Table 2

Results of semi-quantitative X-ray diffractometric analysis of TC and $MnFe_2O_4$/TC

Mineral	Formula	Concentration, %	
		TC	$MnFe_2O_4$/TC
Calcite	$Ca(CO_3)$	29.9	21.4
Quartz	SiO_2	29.9	35.9
Kaolinite	$Al_2(Si_2O_5)(OH)_4$	23.7	
Dolomite	$CaMg(CO_3)_2$		9.5
Chlorite	$(Mg, Fe)_5Al(Si_3Al)O_{10}(OH)_8$	8.2	12.3
Mica	$KAl_2(AlSi_3O_{10})(OH)_2$	5.7	7.3
Feldspars (albite)	$Na(AlSi_3O_8)$	3.4	13.6

The natural clay of the Turkestan deposit was studied by FTIR spectroscopy. Changes in the absorption bands on the spectra of these clays after modification of Mn(II) and Fe(III) were analyzed (Figure 4). The FTIR spectra of all compounds were recorded in solid form in KBr tablets.



a *b*

a — TC; *b* — $MnFe_2O_4$/TC

Figure 4. FTIR spectra

The analysis of the above FTIR spectrum illustrates that the main bands in natural clay relate to the valence bonds of silicon with oxygen and hydrogen with oxygen. The absorption band at *ca.* 1613 and 3637 cm^{-1} corresponding to the deformation vibrations of OH^- groups at the vertices of silicon-oxygen tetrahedra, is a distinctive feature of kaolinites (Fig. 4).

Having characterized the absorption bands on the FTIR spectrum of a sample of clay from the Turkestan deposit, it is possible to implement a similar assignment of the absorption bands on the spectrum of natural clay. The absorption bands at 1433, 2029, and 2517 cm^{-1} allow the identification of calcite ($CaCO_3$) in the clay, which is confirmed by X-ray diffractometric analysis. The FTIR spectrum of albite contains an intense band in the frequency range of 1757-1036 cm^{-1} with a maximum in the region of 1433 cm^{-1} (Fig. 4*a*).

Muscovite mica is an aqueous $KAl_2(AlSi_3O_{10})(OH)_2$. The absorption bands of the SiOSi, SiOAl, Al(OH), and H(OH) groups are usually observed in their spectra. In the clay samples, absorption bands of 457, 794, 1035, and 3695 cm^{-1} are observed, which according to the database corresponds to the mineral muscavite (Fig. 4).

Oxygen atoms can be bound to two silicon atoms by Si–O–Si bridging bonds, or to one by Si–O nonbridging bonds. In particular, the bands 1022 and 1009 cm^{-1} are attributed to the valence vibrations of Si–O–Si(Al) bridging bonds in the crystal lattice. When silicon is partially replaced in the lattice, most of the valence bands of layered minerals (1000–900 cm^{-1}) move in the direction of low frequencies. The appearance of absorption bands in the region of 1000–900 cm^{-1} can be associated with valence vibrations of non–bridge Si–O bonds in various silicate and aluminosilicate groups, as well as in simple ortho-and diorthosilicate anions in the amorphous phase. Aluminum is found in aluminosilicates in either tetrahedral coordination or octahedral coordination. The very weak bands at 794 and 547 cm^{-1} in the samples can be explained by Si–O–Si (Al) bonds in distorted tetrahedral and octahedral layers. In particular, the band in the region of 547cm^{-1} refers to the Si–O–Si strain vibrations involving bridged oxygen, and the band of 794 cm^{-1} refers to the Si–O–Si valence symmetric vibrations characteristic of silicon in the SiO_4 tetrahedron.

Figure 4*b* shows the FTIR spectrum of the modified $MnFe_2O_4$/Turkestan sample. Two strong absorption peaks of 1442 cm^{-1} in the sample indicate the presence of ferrites. The stretching between 400 and 700 cm^{-1} is an oscillation (Fe-O) that indicates the formation of the spinel ferrite structure. In ferrites, metal ions occupy two different interstitial sites in the lattice. One is in a tetrahedral location, and the other is in an octahedral location. From the FTIR spectra, it was found that the high-frequency bands at 875 cm^{-1} are associated with the tetrahedral region, while the low–frequency bands at 629 cm^{-1} are associated with the octahedral region. The sharpness of these bands correlates with the high degree of crystallinity of $MnFe_2O_4$ structures. The wide band of vibrations at 3449 and 3421 cm^{-1} is associated with the stretching vibrations of O–H adsorbed water molecules, indicating a higher amount of surface OH [17].

The absorption bands in the modified clay at 547 and 457 cm^{-1} become more diffuse with a slight change in the position in the spectrum. The 457 cm^{-1} band indicates the presence of Fe–O oscillations, the absence of a band at 621 cm^{-1} (characteristic of the γ-Fe_2O_3 phase), and, at the same time, the appearance of a band at 520 cm^{-1} may indicate a phase transformation of γ-$Fe_2O_3 \rightarrow \alpha$-$Fe_2O_3$ [18].

Conclusions

A method for synthesizing clay composites based on a natural clay of Turkestan (TC) with manganese ferrite has been developed and physico-chemical properties determined. Magnetic adsorbents $MnFe_2O_4$/TC with perfect magnetic separation characteristics were successfully obtained by chemical co-deposition. The clay of the Turkestan deposit in Southern Kazakhstan was studied by physico-chemical methods of analysis: the elemental composition of natural clay and the chemical composition of the modified $MnFe_2O_4$/TC composite and the quantitative ratios of the crystal phases were determined. The study of the FTIR spectra of natural and modified clay deposits of Turkestan allowed us to establish their distinctive features, which consist in the imperfection of their crystal structures. It is expected that the resulting magnetic composite $MnFe_2O_4$/TC can be used as potential catalysts and adsorbents for diverse applications.

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Түркістан кен орнынан алынған табиғи сазбалшық негізінде жаңа $MnFe_2O_4$/сазбалшық магниттік композиттің синтезі және сипаттамасы

Мақала магниттік композиттердің физика-химиялық сипаттамасымен байланысқан табиғи сазбалшықты марганец ферриті негізінде магниттік композиттерді синтездеудің жаңа әдісін жасауға арналған. Зерттеуде магниттік композиттер алу үшін Түркістан кен орнынан алынған Қазақстанның табиғи сазбалшығы пайдаланылды. Магниттік қасиеттері бар материалдарды қалыптастыру биомедициналық жүйелер, электронды құрылғылар, каталитикалық және адсорбциялық процестер үшін магнитті басқарылатын материалдарды әртүрлі қолдану қажеттіліктеріне байланысты біздің заманымыздың өзекті мәселесі. Мұндай материалдардың артықшылығы оларды араластыру, қалпына келтіру, индукциялық қыздыру және т.б. үшін магнит өрісінің көмегімен басқару мүмкіндігі болып табылады. Үлгілер 5 моль/л$^{-1}$ NaOH марганец пен темір тұздарын Түркістан сазбалшығының (ТС) негізінде тұндыру арқылы алынды. Материалдар Фурье-инфрақызыл спектроскопия (FTIR-спектроскопия), рентгендік дифрактометриялық талдау (XRD) және элементтік талдау сияқты әртүрлі талдау әдістерімен сипатталды. РДТ физика-химиялық зерттеулерінің және термиялық талдаудың нәтижелері бойынша ТС құрамындағы негізгі минерал каолинит болып табылады. Магниттік қасиеттері бар $MnFe_2O_4$/Түркістан магниттік адсорбенттері химиялық тұндыру әдісі бойынша сәтті алынды.

Кілт сөздер: табиғи саздар, магниттік материал, марганец ферриті, адсорбент, модифицирленген композит, металл иондары, химиялық қосылыстар, адсорбция.

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Синтез и характеристика нового магнитного композита $MnFe_2O_4$/глина на основе природных глин, полученных из Туркестанского месторождения

Статья посвящена разработке нового метода синтеза магнитных композитов на основе феррита марганца на природной глине с учетом их физико-химической характеристики. В исследовании для получения магнитных композитов использовалась природная глина Казахстана, полученная из Туркестанского месторождения. Формирование материалов с магнитными свойствами является актуальной задачей нашего времени, в связи с потребностями различных применений магнитоуправляемых материалов для биомедицинских систем, электронных устройств, каталитических и адсорбционных процессов. Преимуществом таких материалов является возможность управления ими с помощью магнитного поля для встряхивания, восстановления, индукционного нагрева и др. Образцы были получены авторами путем совместного осаждения солей марганца и железа с 5 моль/л$^{-1}$ NaOH с туркестанской глиной (ТГ). Материалы характеризовались различными методами анализа, такими как Фурье-инфракрасная спектроскопия, рентгеновский дифрактометрический анализ и элементный анализ. По результатам физико-химических исследований РДА и термического анализа основным минералом в составе ТГ является каолинит. Магнитные адсорбенты $MnFe_2O_4$/Туркестан с совершенными характеристиками магнитной сепарации были успешно получены химическим соосаждением.

Ключевые слова: природные глины, магнитный материал, феррит марганца, адсорбент, модифицированный композит, ионы металлов, химическое соосаждение, адсорбция.

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